PROCESS FOR MAKING A PELLET

The present invention relates to a process for making pellets of a thermoplastic extrudable polymer.

Processes for making pellets of thermoplastic extrudable polymer are well known in the plastic industry. Typically the pellets are cylindrical and approximately 3mm in diameter and 3mm in length. The pellets are used in a wide range of plastic article manufacturing processes.

The pellet manufacturing process generally includes a plastification step. In this step the formulation to be pelletised is melted and fed into a twin screw extruder. This has been seen to be beneficial as the pellets produced have been found to comprise of a homogeneous blend of the pellet components due to effective mixing of all molten components in the extruder.

EP-A-0 415 357 describes the making of pellets comprising polyvinylalcohol (PVOH) by melt extrusion with the extrusion being carried out in the temperature range of 150-195°C.

Pelletising processes having a plastification step have several disadvantages associated therewith. The principle disadvantage is the requirement for heating, which means that the energy consumption of these processes is very high.

Furthermore these 'hot' processes are not suitable for polymers which are heat sensitive (such as PVOH) due to heat induced decomposition. Also these 'hot' processes give a heat

history to the polymer which has been found to negatively influence properties of the polymer. In the case of PVOH this has been found to detrimentally affect the PVOH water solubility .

In other pelletising processes dry compaction of the pellet components is carried out at low temperature. Thus the disadvantages of the 'hot' processes are avoided.

WO-A-98/26911 describes a low temperature process for the manufacture of PVOH pellets. In the process the pellets components, in this case a mixture of powdered PVOH and various additives such as plasticisers is fed between two rollers and compressed into pellets. The PVOH component in the pellet blend is not melted in the process and so the issue of heat degradation is avoided.

Also GB-937 057 describes such a low temperature compression process. This follows initial mixing of the plasticiser and PVOH at an elevated temperature.

However, although this process (the cold compression process) eliminates the problem of heat induced decomposition of the polymer, the pellets produced suffer from other disadvantages.

Most of the disadvantages stem from the inherent nature of the compaction process, more specifically the rollers and the powder feed thereto. It has been found to be very difficult to ensure that the powder feed is spread evenly across the rollers. This has the effect that control of the

size of the pellets is difficult and so the size of the pellets can vary significantly.

Furthermore significant dust formation is typical for this kind of process. Additionally the pellets are commonly friable having poor integrity and easily form dust from friction rubbing against each other, thus worsening the dust issue. Both of these issues are attributed to the poor spreading and roller compression technique.

Furthermore significant variability of the composition of the pellets and poor homogeneity of the pellets has also been observed. The issues are also believed to be associated with the poor powder distribution over the rollers. The problem of the variability of the pellet composition and the poor homogeneity of the pellets is exacerbated when the pellets are taken and used in a further processing step.

These kinds of pellets, wherein the thermoplastic polymer component of the pellets is PVOH, are used in the manufacture of water soluble PVOH pouches in extrusion / injection moulding processes. The pouches, as an example, are commonly used to contain a detergent composition for use in an automatic washing machine (laundry / dishwasher). In these applications is it vital that the pellets have high homogeneity to ensure that the pouches produced have good integrity to be stable in storage and have the expected water dissolution properties.

Pellets produced in a cold compaction process, as described above, often fail to meet the level of homogeneity required for the processing into the pouch format.

It is an object of the present invention to obviate / mitigate the problems outlined above.

According to the present invention there is provided a shaping process for making pellets of a thermoplastic extrudable resin composition comprising a thermoplastic polymer, plasticiser and optionally further additives, the plasticiser comprising a component which is solid at room temperature, wherein the process is run at a temperature above the melting point of the plasticiser and below the melting / plastification temperature of the thermoplastic polymer.

The shaping process may comprise pressing, extrusion, calendering and / or compaction. Most preferably the shaping process comprises extrusion.

The process of the present invention has been found to overcome the disadvantages associated with the prior art. Firstly as the process is operated at a temperature below the melting / plastification temperature of the thermoplastic polymer the process has been found to be extremely energy efficient. Furthermore the heat degradation of heat sensitive materials in the resin blend is dramatically reduced by the lowered process temperatures.

Additionally as the process operates above the melting point of the plasticiser (which is then allowed to cool to form

the solid pellet) the pellets have been found to have a very low friability. Thus the pellets have a much lower tendency to release dust upon friction rubbing.

Furthermore as the pellets are produced at a temperature above the melting point of the plasticiser component the pellets have been found to have excellent homogeneity. More specifically both the overall composition of each pellet and the distribution of the individual components within the pellets have been found to have an high level of predictability and low variance. This is especially important when the pellets are used in a further processing step such as a second extrusion process (e.g. injection moulding) for the manufacture of an article comprising the thermoplastic polymer.

Generally the components are delivered to the shaping equipment used in the process in particulate form.

It has been found that he particle size of the raw materials used to make the pellets should be small. This has been observed to ensure high homogeneity of the pellets. The particle size of the raw materials used preferably is below 2000µm, more preferably below 1200µm, more preferably below 400µm and most preferably about 200µm.

Preferably the plasticiser is present in the composition with at least 5%, more preferably 10%, most preferably 15%.

Preferably the temperature of the material within the extruder does not exceed a temperature which is 10°C below

the melting / plastification temperature of the thermoplastic polymer at any time. More preferably it does not exceed 15°C, more preferably 30°C and most preferably 45°C below the melting / plastification temperature of the thermoplastic polymer. However, it is desired that the temperature of the material exceeds the ambient air temperature. Preferably the temperature of material within the extruder is at least 40°C, more preferably at least 45°C, and most preferably at least 50°C.

The plasticiser has to at least partially melt at the preferred operating temperature. The melting point of the plasticiser component is preferably at least 15°C, preferably at least 30°C and most preferably at least 45°C below the melting / plastification temperature of the thermoplastic polymer.

Preferably the plasticiser comprises a carbohydrate.

Carbohydrates are usually represented by the generalised formula $C_x(H_2O)_y$. The term herein also includes materials which are similar in nature like gluconic acids or amino sugars which cannot be fully represented by this formula. Other carbohydrate derivatives like sugar alcohols such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt fall within this term.

Most preferred carbohydrates include the more thermally stable carbohydrates such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt.

Other preferred plasticiser systems include solid fatty acid alkoxylates, fatty alcohol alkoxylates or polyalkylene glycols (such as long chain polyethylene glycol).

The plasticiser may comprise a further auxilliary component. Preferred auxilliary components include glycerin, ethylene glycol, propylene glycol, diethylene glycol, diproylene glycol, triethanol amine, diethanol amine and methyldiethyl amine.

Once the or each strand has issued from the extruder it may be permitted to cool under ambient conditions. Alternatively cooling may be assisted. One way in which this may be done is by employing a cooled metal belt onto which the or each strand issues. Another way in which this may be done is by using a cooled fluid, preferably cooled air, downstream of the extruder. Another way is by blowing a fluid, preferably air, across the or each strand. One or more of these methods may be used.

Preferably the or each strand is separated into pellets, during the manufacture.

The strands are separated into pellets preferably by cutting. However, other separation methods, for example twisting, are not ruled out. A method may be envisaged whereby the strand is twisted at intervals when still plastic, to form "sausages", which can be separated by breaking the connections, once they have become more brittle. Partial cutting or pressing or nipping or perforating (all such methods

collectively called "scoring" herein) to form frangible separation webs, may also be employed, to form tablet precursors. Separation of the precursors to produce pellets may be effected during manufacture or by the consumer, manageable lengths being provided from which the consumer breaks or twists off pellets as required. A pellet precursor may be, for example, a straight row of pellets, to be broken off as needed.

The extrusion pressure may be whatever is required to carry out the process in an efficient manner. Suitably it is in excess of 3 bar (0.3MPa), preferably in excess of 5 bar (0.5MPa), and more preferably is preferably in excess of 8 bar (0.8MPa). More preferably still is preferably in excess of 12 bar (1.2MPa). Most preferably it is in excess of 40 bar (4MPa). The extrusion pressure preferably does not exceed 100 bar (10MPa), more preferably 60 bar (6MPa).

Generally the pellets are for use in injection moulding processes. The injection moulding process is preferably used for the manufacture of water soluble pouches intended to contain a detergent formulation for use in an automatic washing machine or in an automatic dishwasher. Thus the pellets preferably comprising a water-soluble / water-dispersible thermoplastic polymer

In this use the advantageous properties of the pellets produced in accordance with the invention, especially the high homogeneity have been found to be particularly beneficial. It is believed that this property is most beneficial as the integrity of the injection moulded product relies upon such

high homogeneity of the composition being injection moulded as otherwise the low homogeneity will be reflected in the injection moulded product. The high homogeneity has been found to lead to predictable water solubility of injection moulded products.

Preferably the water-soluble / water-dispersible thermoplastic polymer comprises PVOH or a derivative thereof.

Other water-soluble / water-dispersible polymers may be used in the process either as an alternative or in addition to PVOH. Preferred examples include poly(vinylpyrollidone), poly(acrylic acid), poly(maleic acid), a cellulose derivative (such as a cellulose ether / hydroxypropyl methyl cellulose), poly(glycolide), poly(glycolic acid), poly(lactides), poly (lactic acid) and copolymers thereof.

Processing aids may be present in the admixture which is processed. Preferred processing aids include mono-, di-, tri-carboxylic acids / salts thereof, fatty acids such as stearic acid / salts thereof, mono-, di- or triglycerides / salts thereof, aerosil, inorganic and organic pigments.

The invention will now be illustrated with reference to the following non-limiting Examples.

Examples:

Example 1:

The pelletising process was conducted on an extruder (twin screw, ICMA S. Giorgio, Milan (dedicated to processing of plastic blends and alloys).

The extruder had the following characteristics.

Screw diameter:	35 mm
Screw length:	40 cm
Working pressure:	30 bar
Output:	5 kg/h.
Temperature zones:	6 (T1=50°C, T2=60°C, T3=T4=90°C, T5=105°C and T6 (the die) =105°C.)

The extruder was attached to a two-roll unit used as a cooling source and connected to a pellet cutter.

The following formula was fed into the extruder in powder form.

Material	%
PVOH resin	85.0
Sorbitol	11.0
Processing aids	4.0
Total	100.0

The pellets obtained were chilled to room temperature. The formula yielded solid pellets having low friability.

Example 2:

The pelletising process was conducted on a pellet press (model V3-75 from Universal Milling Technologies).

The press had the following characteristics.

Die diameter:	350 mm
Holes diameter:	2 mm
Hole length:	3 mm
Infeed cone:	45°
Space between die / rollers:	1.5 mm
Die speed:	5m/s
Motor:	30 kW
Temperature:	98-102°C

The following formulae were fed into the extruder in powder form.

Material	Formula 1	Formula 2	Formula 3
PVOH resin	81.0	87.0	85.0
Sorbitol	15.0	11.0	11.0
Processing aids	4.0	2.0	4.0
Total	100.0	100.0	100.0

The pellets obtained were chilled to room temperature. Each formula yielded solid pellets having low friability.